

High intensity ultrasound-assisted extraction of oil from soybeans

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Abstract

The application of 20 kHz high-intensity ultrasound during extraction of oil from two varieties of soybeans (TN 96-58 and N 98-4573) using hexane, isopropanol and a 3:2 hexane–isopropanol mixture was evaluated. In a simplified extraction procedure, ground soybeans were added to solvents and ultrasonicated between 0 and 3 h at ultrasonic intensity levels ranging from 16.4 to 47.6 W/cm². Oil was recovered after distillation and yield and composition determined. Using hexane as a solvent, yield generally increased as both application time and intensity of ultrasound increased. Solvent type influenced the efficiency of the extraction, i.e., the highest yield was obtained using ultrasound in combination with the mixed solvent. Gas chromatography analysis of ultrasonicated soybean oil did not show significant changes in fatty acid composition. Results were attributed to mechanical effects due to ultrasonically induced cavitation increasing permeability of plant tissues. A comparison of scanning electron microscopy images of raw and ultrasonicated soybeans indicated development of microfractures and disruption of cell walls in ground soybean flakes. Our study suggests that high-intensity ultrasound may reduce time required to extract edible oils from plant sources and hence improve throughput in commercial oil production processes.

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1. Introduction

Plant-based lipophilic compounds such as edible oils, phytochemicals, flavors, fragrances and colors are valuable products in the food, pharmaceutical and chemical industry. Extraction is one of the key processing steps in recovering and purifying lipophilic ingredients contained in plant-based materials (Liu, 1999). Classical extraction technologies are based on the use of an appropriate solvent to remove lipophilic compounds from the interior of plant tissues. The choice of a suitable solvent in combination with sufficient mechanical agitation influences mass transport processes and subsequently efficiency of the extraction. The most widely used solvent to extract edible oils from plant sources is hexane. Hexane is available at low cost and is efficient in terms of oil and

solvent recovery (Mustakas, 1980; Serrato, 1981). More recently, the use of alternative solvents such as alcohols (isopropanol or ethanol) and supercritical carbon dioxide has increased due to environmental, health and safety concerns (Dunnuck, 1991). Alternative solvents are often less efficient due to a decreased molecular affinity between solvent and solute and costs for solvent and process equipment can be higher (Baker & Sullivan, 1983; Freidrich & Pryde, 1984; Karnofsky, 1981).

A potential new technology that may improve extraction of lipophilic compounds from plants is high-intensity ultrasound. High-intensity ultrasonication can accelerate heat and mass transport in a variety of food process operations and has been successfully used to improve drying, mixing, homogenization and extraction (Fairbanks, 2001; Mason, 1992; Mason, Paniwnyka, & Lorimera, 1996; Povey, 1998). Ultrasonication is the application of high-intensity, high-frequency sound waves and their interaction with materials (Luque-García & Luque de Castro, 2003). The propagation and

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interaction of sound waves alters the physical and chemical properties of materials that are subjected to ultrasound (Mason & Lorimer, 1988). In the case of raw plant tissues, ultrasound has been suggested to disrupt plant cell walls thereby facilitating the release of extractable compounds and enhance mass transport of solvent from the continuous phase into plant cells (Vinatoru, 2001).

Hui, Etsuzo, and Masao (1994) utilized ultrasound to extract saponin from ginseng and observed that yield of total extraction increased by 15% and yield of saponin by 30%. Romdhane and Gourdon (2002) investigated extraction of pyrethrins from pyrethrum flowers and oil from woad seeds. In both cases, acceleration of extraction kinetics and increase in yield was observed, however less so in the case of woad seeds. Vinatoru et al. (1997) showed improved yields of lipophilic compounds extracted from herbs such as coriander and fennel.

Based on these studies, we hypothesize that application of high-intensity ultrasound may improve extraction of oil from soybeans. The objective of this study was to test this hypothesis by determining the influence of sonication time and intensity in combination with different solvents on the efficiency of oil extraction from soybeans.

2. Materials and methods

2.1. Materials

Two soybean varieties, TN 96-58, a popular Tennessee variety, and N 98-4573, a North Carolina specialty variety, were obtained from the Crops Laboratory at The University of Tennessee. Compositional analysis of the two soybean varieties indicated a total lipid content of 19.6% for TN 96-58 and 19.1% for N 98-4573, a protein content of 42.2% for TN 96-58 and 42.7% for N 98-4573 and an ash content of 5.43% for TN 96-58 and 5.34% for N 98-4573 (Stassi, 2003). AOCS Mix No. 3, a fatty acid standard for GC analysis, was purchased from Alltech Corporation (Deerfield, IL, USA) and kept in a refrigerator at 4 °C until analysis. Hexane and isopropanol (99.8% purity) were purchased from Fisher Scientific (Fair Lawn, NJ, USA).

2.2. Methods

2.2.1. Soybean flake preparation

Raw soybeans were cleaned using a grading procedure established by the Federal Grain Inspection Service (FGIS, 1997) to remove any foreign material such as small stones, sand and plant leaves that may be present after harvesting, drying, transportation and storage. Soybeans (125 g) were sieved and soybeans larger than 3.18 cm ($\frac{8}{64}$ in.) were collected. The cleaned, raw soy-

beans (moisture content approx. 8% w.b.) were stored in an environmental chamber containing potassium iodide solution (69.9% relative humidity at 22 °C) to adjust their moisture content to the optimal value suitable for subsequent grinding and extraction (Liu, 1999). Moisture content of soybeans was recorded every two hours using a single kernel moisture tester (CRT-160E, Shizuoka Seiki, Japan) until a final moisture content of 11% was reached. Cleaned and conditioned soybeans were ground using a hammer mill (Standard Model No. 3, Arthur Thomas Co., Philadelphia, PA, USA) running at 478 RPM. A stainless steel screen with a mesh size of 4 mm was used to obtain a consistent particle size distribution of soybean flakes (Fig. 1). Ground soybean flakes were then packaged in air-tight plastic bags until used.

2.2.2. Sonication and extraction procedure

Ground soybean flakes (100 g) were mixed with 150 ml solvent in a 600 ml plastic beaker. The soybean-solvent suspension was ultrasonicated for 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 h using a 20 kHz ultrasonic generator (S3000, Misonix Incorporated, Farmingdale, NY, USA) with a 1.27 cm probe that was submerged in the suspension. Ultrasonic wave intensities were determined calorimetrically (Eq. (3)) and ranged from 16.4 to 47.6 W/cm². Suspensions were kept in a waterbath at 25 °C during sonication and extraction. Suspensions were continuously stirred at a constant stirring rate using a magnetic stirrer to prevent heating of suspensions under the influence of high-intensity ultrasound. Controls included soybean flakes that were extracted using the same solvent without applying ultrasound. After extraction, oil was separated from the solvent-soybean suspension using a countercurrent distillation set-up with the heat source set to 110 ± 5 °C and water as the coolant (Li, 1999).

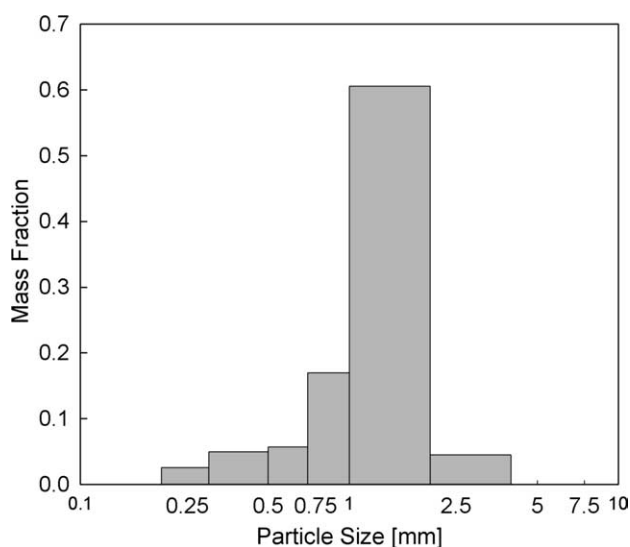


Fig. 1. Particle size distribution of ground soybeans.

2.2.3. Yield determination

Extraction yield was determined gravimetrically as

$$Y = \frac{m_e/m_t}{m_l/m_t} = \frac{x_{el}}{x_{tl}}, \quad (1)$$

where m_e is the mass of extracted lipids (g), m_t the ground soybean weight (g), m_l the total lipid mass of the soybean flakes (g), x_{el} the extracted lipid fraction and x_{tl} the total lipid fraction of soybeans (19.6% and 19.1% for TN 96-58 and N 98-4573, respectively).

2.2.4. Calorimetric determination of ultrasonic wave intensities

The intensity of the generated ultrasonic wave was determined using a calorimetric method (Mason et al., 1996). For each suspension, the temperature T was recorded with a thermocouple as a function of time under adiabatic conditions. From temperature versus time data, the initial temperature rise dT/dt was determined by polynomial curve fitting. The absolute ultrasonic power P was calculated as

$$P = mc_p \left(\frac{dT}{dt} \right), \quad (2)$$

where m is the total mass and c_p is the heat capacity of the solvent. The intensity of ultrasonic power dissipated from a probe tip with radius r is given by

$$I = \frac{P}{\pi r^2}. \quad (3)$$

For input power levels of 90, 120 and 180 W, the calculated intensities were 16.4, 20.9 and 47.6 W/cm², respectively.

2.2.5. Fatty acid profile determination by GC

Fatty acid (FA) profiles were determined according to the AOCS official methods that describe preparation of FAME (Ce 2-66) and GC analysis (Ce 1-62) (AOCS, 1998). FA profile determination included extraction of lipid samples with organic solvents, followed by transformation of the isolated lipid to fatty acid methyl esters (FAME) and quantification of FAME by gas chromatography. FA profiles were analyzed using a Hewlett-Packard 6890 gas chromatograph with cold on-column injection in a capillary column (HP-2980 (30 m × 0.25 mm × 0.1 μm)) and by flame ionization detection. Injection temperature was set at 130 °C, rising at 3 °C/min to 210 °C with a 10 min holding time and a detector temperature of 250 °C. Helium carrier-gas column flow rate was 1.8 ml/min with a make-up gas flow rate of 30 ml/min. The flow rate of hydrogen and air was 40 ml/min and 400 ml/min, respectively. Prepared FAME (2 μl) was introduced into the GC with a split ratio of 1:10. The ratio of unsaturated fatty acid to saturated fatty acid content was used as an indicator for soybean oil compositional changes.

2.2.6. Electron microscopy

An in-lens field emission scanning electron microscope (S-3500N, Hitachi SEM) was used at an operating voltage of 20 kV at a vacuum of 15 Pa. High resolution topographic images at low (100×), medium (1000×) and high (4000×) magnifications were digitally recorded with short dwell times to prevent beam induced damage. Samples were deposited on a silicon wafer and coated with a conductive material (gold) to ensure sufficient electron refraction.

2.2.7. Statistical analysis

Duplicate samples were used. All measurements were conducted in triplicates. Least square means were analyzed using the general linear model of the Statistical Analysis System (SAS Institute, Cary, NC).

3. Results and discussion

3.1. Solvent extraction in the absence of high-intensity ultrasound

The oil extraction capabilities of three different solvents (hexane, isopropanol, and hexane:isopropanol mixture, 60:40%, v/v) at extraction times ranging from 30 min to 3 h are shown in Fig. 2. When the extraction time increased from 30 min to 3 h oil yield of TN 96-58 increased by 4.5%, 5.8% and 8.8% using isopropanol, hexane, and the mixed solvent. In general, oil yield increased with treatment time irrespective of the type of solvent used, but the mixed solvent was superior in terms of oil yield increase (approx. 9%) when compared

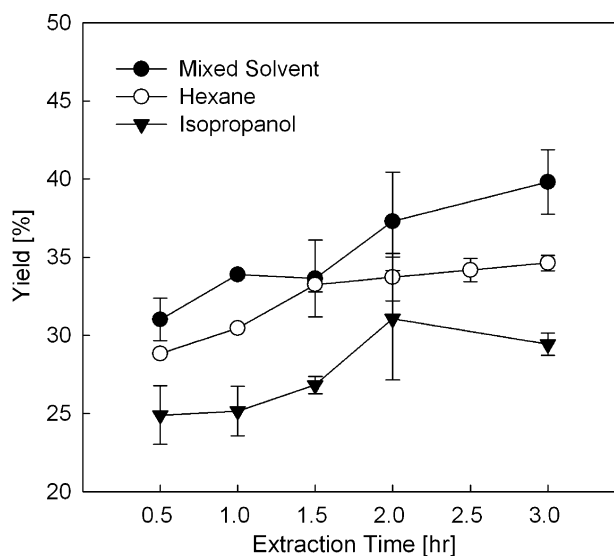


Fig. 2. Oil yield as a function of extraction time for soybean variety TN 96-58 using hexane, isopropanol and hexane:isopropanol as solvents at 25 °C.

to the efficacy of hexane or isopropanol. After 30 min using the mixed solvent, oil yield was 3.9% higher than that of hexane, which in turn was 2.2% higher than that of isopropanol. When the treatment time was increased to 3 h, the oil yield using the mixed solvent was 5.2% higher than that of hexane, which was 5.2% higher than that of isopropanol.

Our results indicate that the efficiency of the extraction process is a function of the molecular affinity between solvent and solute in agreement with earlier studies (Meniai & Newsham, 1992). The higher efficiency of the isopropanol:hexane mixture has previously been reported by Hara and Radin (1978) in a lipid extraction experiment using rat and mouse tissue and more recently by Schäfer (1998) who extracted cereal lipids using a 2:3 isopropanol:hexane mixed solvent. It should be noted that the overall extraction efficiency of our simplified extraction method after 3 h was low (absolute oil yields: 34.6% for hexane, 20.4% for isopropanol and 39.8% for hexane:isopropanol). This may be attributed to the fact that: (a) hulls were not removed in our simplified extraction procedure as is often practiced commercially, (b) the asymptotic final yield may only be obtained after significantly longer extraction times and (c) the use of a hammer mill instead of a flaking roll may yield non-optimal particle sizes. Thus higher yields may be obtained in a commercial process.

3.2. Influence of ultrasonic wave intensity on oil yield

The influence of different ultrasound intensity levels (16.4, 20.9, and 47.6 W/cm²) on oil yield is shown in Fig. 3. Oil yield increased with increasing ultrasonic intensity. After 3 h at an ultrasound intensity of 47.6 W/cm², the increase in oil yield was 2.4% higher than at an ultrasonic intensity of 20.9 W/cm² and 9% higher than at 16.4 W/cm² (Fig. 3). Compared to the nonsonicated control, the oil yield after 3 h at 16.4, 20.9 and 47.6 W/cm² increased by 2.2%, 10.1% and 11.2% respectively. Thus, after three hours, the relative oil yield increase at 47.6 W/cm² was approximately five times higher than at 16.4 W/cm².

Improved soybean oils yields may be explained in terms of cavitation effects caused by the application of high-intensity ultrasound. As large amplitude ultrasound waves travel through a mass medium, they cause compression and shearing of solvent molecules resulting in localized changes in density and elastic modulus (Price, White, & Clifton, 1995). As a consequence, the initially sinusoidal compression and shear waves will at a finite distance from the ultrasonic transducer be distorted into shock waves. The abrupt decrease in pressure at the edge of the saw tooth shaped ultrasonic wave in the negative pressure cycle generates small bubbles. These bubbles collapse in the positive pressure cycle and produce turbulent flow conditions associated with high

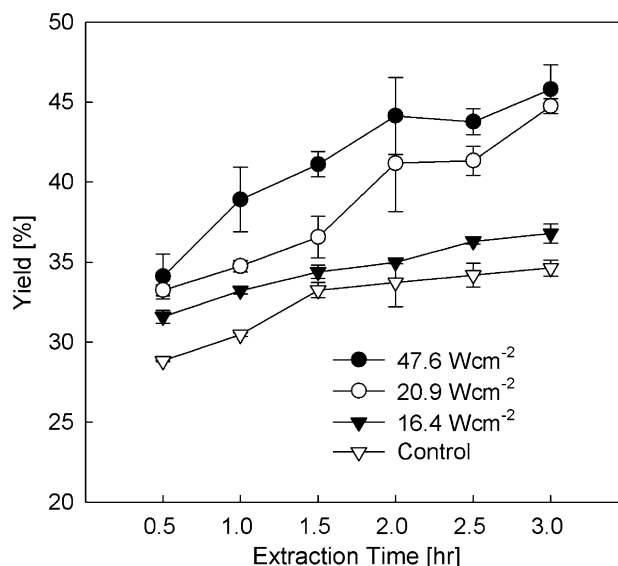


Fig. 3. Oil yield as a function of extraction time for soybean variety TN 96-58 using high-intensity ultrasound at ultrasonic intensities of 0, 16.4, 20.9 and 47.6 W/cm² using hexane as a solvent at 25 °C.

pressures and temperatures (Mason, 1997; Mason & Cordmas, 1996; Mason, 1992; Price, 1990, 1993). Since formation and collapse of bubbles occurs over very short periods of time, typically a few microseconds (Hardcastle et al., 2000), heat transfer from cavitation bubbles to the medium is small causing only gradual temperature increases in the medium. Therefore, decreases in solvent viscosity are small and are most likely not the principal cause of the yield increases. Rather, at increasing amplitudes, cavitation bubble collapse is more violent since the resonant bubble size is proportional to the amplitude of the ultrasonic wave (Suslick, Casadonte, Green, & Thompson, 1987; Suslick & Price, 1999). Bubble collapse in the vicinity of plant membranes may cause strong shear forces to be exerted that can cause microfractures to be formed in plant tissues (Vinatoru, 2001; Vinatoru et al., 1997).

Fig. 6 shows a set of SEM images of TN 96-58 soybean flakes at a magnification factor of 1000× (a) after 3 h of conventional hexane extraction, (b) 1 h of ultrasound-assisted hexane extraction and (d) 3 h of hexane assisted extraction. Microfractures appeared in the soybean flakes after application of ultrasound for 1 h (Fig. 6(b)) and the surface morphology of soybean flakes visibly changed after 2 h of sonication (Fig. 6(c)) that is the soybean flake surfaces became more porous.

3.3. Influence of soybean varieties on ultrasound-assisted extraction of soybean oil

The oil yield of both varieties of soybeans increased with application of ultrasound (Fig. 4) but the relative increase in oil yield of the two soybean varieties with extraction time differed. For the TN 96-58 variety, the yield

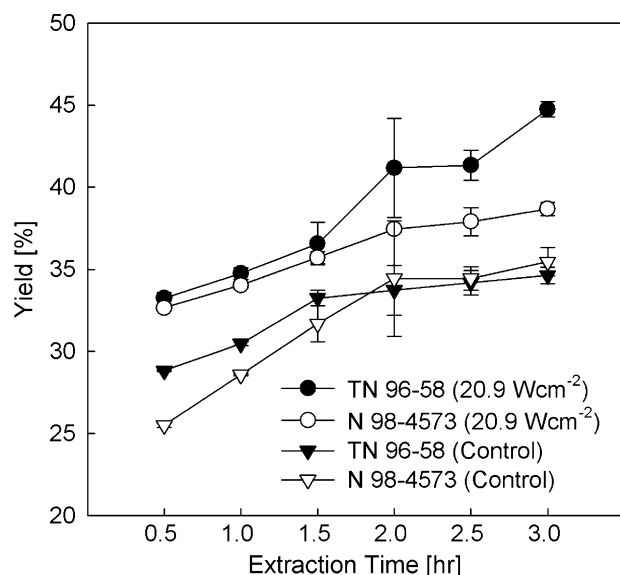


Fig. 4. Oil yield as a function of extraction time for soybean varieties TN 96-58 and N 98-4573 treated with ultrasound at an intensity of 20.9 W/cm² using hexane as a solvent at 25 °C.

increased by 4.4% between the control group and the 20.9 W/cm² ultrasound-assisted group at a reaction time of 30 min to reach a yield difference of 9.4% after 3 h. For this variety, ultrasound had a more pronounced effect on the yield in the latter stage of the extraction. In contrast, for N 98-4573, the oil yield difference between the control and the ultrasound-assisted group was 7.1% after extraction/sonication for 30 min and increased only by another 3.2% after 3 h. For this variety, ultrasound enhanced oil yield particularly in the early stage of the extraction process.

Results shown in Fig. 4 may be related to difference in soybean structure (Romdhane & Gourdon, 2002). As noted by Romdhane and Gourdon (2002), the rheological nature of the seed structure (hardness, compactness) may have a direct impact on the capability of ultrasound to improve extraction of lipid compounds from plant cells. While a compositional analysis of the two soybean varieties showed little difference between the two varieties in protein content (42.7% for N 98-4573 and 42.2% for TN 96-56), ash content (N 98-4573: 5.34%; TN 96-56: 5.43%) and total lipid content (N 98-4573: 19.1%; TN 96-56: 19.6%), a more in-depth analysis of the cell wall structure may help explain the exact nature of the observed differences between the two plant varieties.

3.4. Influence of molecular properties of solvents on ultrasound-assisted extraction of soybean oil

The difference between oil yield obtained with hexane and isopropanol as solvents after 30 min using the classical extraction process was 3.9% (Fig. 5). When the

reaction time was increased to 3 h, the difference in yield increased slightly to 5.2%. Comparison of the relationship between yield and extraction time for the classical extraction using different solvents illustrates that the selection of solvent influences oil yield. In the case of ultrasound enhanced extraction using pure hexane and isopropanol, the difference between yields was less pronounced. After 30 min, the oil yield using hexane was 2.4% higher than with isopropanol. When the reaction time was increased to 3 h, the oil yield obtained with isopropanol was 1.1% higher than with hexane as a solvent. The difference between the ultrasound-assisted and the control group after 30 min of extraction using hexane was 4.4% while the difference between ultrasonicated and untreated soybeans using isopropanol was 5.9%. At a reaction time of 3 h, the difference increased to 10.1% and 16.4%, respectively. It is apparent in Fig. 5 that in the ultrasound-assisted extraction operation there was a greater increase in oil yield when isopropanol was used as a solvent than when hexane was used.

A solvent mixture was prepared by mixing hexane and isopropanol at a ratio of 60:40% (v/v). Oil yields obtained with all three solvents (hexane, isopropanol and the solvent mixture) both with and without ultrasound assistance are shown in Fig. 5. The mixed solvent clearly had a much better extraction performance than any of the other solvents. At an extraction time of 30 min, the oil yield using the mixed solvent was 2.2% higher than with hexane and 6.1% higher than with isopropanol. When the reaction time was increased to 3 h, the oil yield using the mixed solvent group increased by 5.2% and 10.4% when compared to hexane and isopropanol, respectively. The extraction capability of the mixed solvent was further

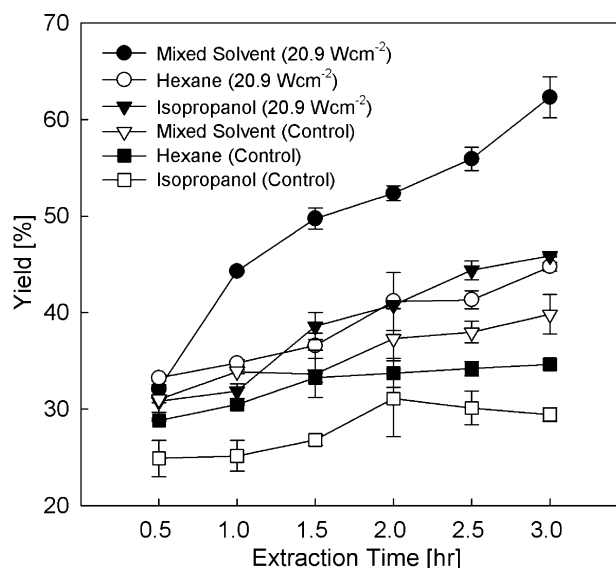


Fig. 5. Oil yield increase of soybean variety TN 96-58 as a function of extraction time using hexane:isopropanol mixture, hexane and isopropanol and treated with (20.9 W/cm²) and without ultrasound at 25 °C.

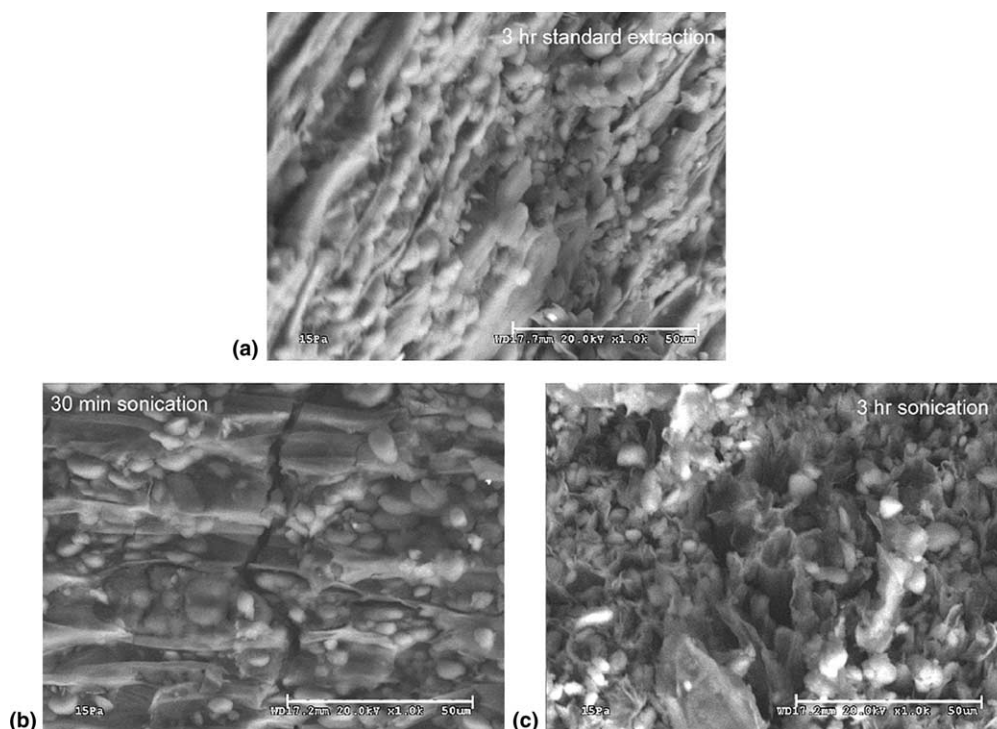


Fig. 6. Scanning electron microscopy images of soybean flakes after (a) 3 h classical hexane extraction, (b) 30 min ultrasound-assisted extraction and (c) 3 h ultrasound-assisted extraction.

enhanced by the application of ultrasound. The absolute oil yield was 32.0% after 30 min and increased almost twofold to 62.3% after 3 h when used in combination with ultrasound. The difference between the ultrasound-assisted group and the control group was only 1.0% at the beginning of the extraction. However, when a reaction time of 3 h was used, the difference increased to 22.5%. These results indicate that for the mixed solvent, reaction time is an important processing parameter affecting the oil yield.

Cavitation in a liquid continuous phase is impacted by the physical properties of the solvent. At 25°C, isopropanol has a vapor pressure of 43 mbar, a viscosity of 2.27 mPa·s, a density of 0.785 g/cm³ and a surface tension of 21.7 mN/m while hexane has vapor pressure of 266 mbar, a viscosity of 0.31 mPa·s, a density of 0.664 g/cm³ and a surface tension of 18.4 mN/m. Chivate and Pandit (1995) demonstrated for binary mixtures of ethanol and water that vapor pressure and surface tension are the two key factors that impact the cavitation intensity at a specific distance from the horn generator, i.e. cavitation intensity decreases as vapor pressure and surface tension increases. While the surface tension of the two solvents does not differ significantly, the vapor pressure of hexane is approximately five times higher than that of isopropanol. As previously stated, solvent affinity between oil and the mixed solvent is higher than for hexane or isopropanol (Hara & Radin, 1978; Schäfer, 1998). Results may thus be attributed to solvent–solute affinity and cavitation

phenomena. Nevertheless additional studies will be required to quantify the contribution of the individual effects of high-intensity ultrasound and solvent on oil yield and to gain a better understanding of the mechanism of ultrasonication.

3.5. FA analysis of ultrasonically extracted soybean oil

Results of the GC analysis of sonicated and untreated soybean oil show a small decrease in the relative percentage of unsaturated fatty acids and an increase in the percentage of saturated fatty acids when ultrasound-assisted extraction was used (Table 1). This ratio is used as an indicator of the extent of fat deterioration because unsaturated fatty acids are more susceptible to oxidation, whereas saturated fatty acids are more stable to oxidation. In the control group, the C18:1/C16:0 ratio was 1.54 while in ultrasound-assisted extraction group it decreased to 1.49. The oxidation percentage was 3.4%. The ratio of C18:2/C16:0 was 5.08 and 5.05 in the control and ultrasound-assisted group, respectively. A difference of 0.52% in the linoleic acid content was observed. Results would indicate that oxidation of soybean oil does occur upon application of ultrasound, however the difference in the GC analysis between the ultrasonicated and the control group was small suggesting that ultrasonication did not noticeably influence composition of the extracted oil.

Table 1

Comparison of ratio of unsaturated to saturated fatty acid (C18:2/C16:0, C18:1/C16:0) of oil extracted for 3 h from TN 96-58 using hexane as a solvent with and without high-intensity ultrasound (47.6 W/cm²)

Retention time (minutes)	Ultrasound-assisted (U)		Control group (C)		U/C ratio
	Peak area	Ratio	Peak area	Ratio	
8.02 ± 0.01 (16:0)	130.01	1	128.29	1	100.00
11.92 ± 0.02	38.6	0.2969	39.52	0.3080	96.40
12.35 ± 0.02 (18:1)	193.11	1.4852	197.27	1.5376	96.59
12.49 ± 0.02	15.96	0.1228	16.03	0.1250	98.24
13.34 ± 0.05 (18:2)	656.48	5.0491	651.14	5.0756	99.48
14.82 ± 0.02	90.46	0.6957	89.7	0.6992	99.50
16.46 ± 0.02	3.6	0.0277	3.83	0.0299	92.64
16.90 ± 0.02	2.4	0.01855	2.4	0.0187	99.20
22.33 ± 0.03	4.5	0.03461	4.77	0.0372	93.04

4. Conclusions

The results obtained in this study have implications for the edible oil industry. Ultrasound has the potential to be used in oil extraction processes to improve efficiency and reduce processing time. During commercial solvent extraction, a series of time-consuming preparation steps is necessary to achieve the maximum oil yield. These key steps involve cleaning, dehulling, moisture conditioning, flaking and heating. Our study demonstrated that a simplified, short term extraction procedure that utilizes ultrasound during the extraction process may be sufficient to obtain commercially acceptable yields. Careful consideration should be given to the choice of an appropriate solvent. The influence of the molecular affinity between solvent and solute is not the only parameter that impacts the suitability of solvent as is the case in classical extraction technologies. Factors that impact cavitation such as solvent vapor pressure and surface tension need to be considered as well.

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